# 國立聯合大學「高等教育深耕計畫」 補助計畫精簡成果報告書

計畫名稱:建立在地特色研究強化跨領域產學鏈結

執行期間: 110 年 06 月 01 日至 110 年 12 月 31 日

執行單位:智慧綠能研究中心

計畫主持人: 陳建仲

處理方式:公開於校網頁

中華民國110年11月30日

1

## 一、 中文摘要:

紹合金廣泛應用於需要有良好的成形性、高抗蝕性、且強度要求不高的產品,例如化工設備、民生生活用品、生醫材料、半導體設備、風力發電機、太陽能電池支架、大型公共建設等。陽極處理目前在中國一年約有 100 億美金的市場需求,在美國一年的市場需求量約為 650 億美金。本研究團隊洞察高科技產業及綠能產業設備表面處理技術的市場需求,並配合聯合大學在地化特色產業與人才培育政策,規劃陽極處理與審製化成品製作,建立具有洽接國際訂單水平的陽極處理技術與人才培育平台,可提供國內廠商推廣新一代陽極處理技術於高附加價值之產品開發與製造上,並做為陽極處理創新產業人才培育之基地。本研究利用直流陽極處理製程於 6061 鋁合金表面成長一層高品質的陽極膜,研究方法包含:(a)陽極前處理、(b)陽極處理、(c)陽極後處理,其中,陽極方法包含:銀合金基材退火應力消除、試片尺寸裁減、表面研磨、表面提出含:銀合金基材退火應力消除、試片尺寸裁減、表面研磨、表面提出,以應力消除、試片尺寸裁減、表面研磨、表面,

## 二、 執行內容:

本研究的陽極處理法製作步驟包括:鋁合金基材退火應力消除、表面研磨、表面拋光、陽極處理、染色、封孔、表面拋光、雷射雕刻、與電泳等,如圖 1 所示。陽極處理所需控制的參數包含電解液成分、電解液溫度、電壓值、電流密度值、與陽極處理時間等。

鋁陽極處理的主要反應式為  $A1^{3+}$  +  $30H^-$  →  $A1(0H)_3$  ,根據法拉第定律(Faraday's laws),陽極處理電流值與膜厚的關係式可表示如式(1),並簡化成式(2)與式(3)

$$Q=It=nFN=nF \rho DA/M$$
 (1)

$$Q=It= nFN= nF \rho DA/M$$
 (2)

$$D=ItM/nF \rho A$$
 (3)

其中Q:電量(C)、I:電流(A)、T:時間(sec)、n:解離價數、F:法拉第常數(96500)、N:莫爾數(mole)、 $\rho$ :密度(g/cm³)、D 厚度(cm)、A:面積(cm²)、M:分子量(g/mol).

根據式(1) n=3;  $\rho$  =2.4g/cm<sup>2</sup>; M=78 g/mo1.

本實驗陽極處理時間為 1200 秒、陽極處理面積為  $50 \text{cm}^2$ ,因此式(3) 可簡化為式(4)

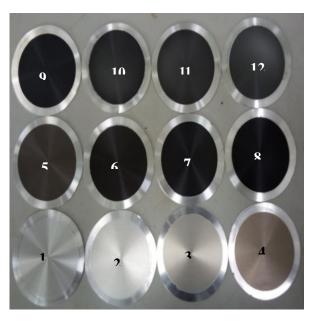
$$D(\mu m) = 26.94 \text{ x I}$$
 (4)

陽極處理效率可進一步計算如下式

其中 Da 為實際量測的陽極膜厚度, D 為理論陽極膜厚度。

本實驗陽極處理實驗參數為陽極處理時間:20 min、陽極電流密度:0.5~12A/dm²、陽極處理樣品面積:50cm²,因此,由電源供應器輸入至陽極處理槽的電量(Q=It)。圖2顯示6061 鋁合金在22℃陽極處理液下通入不同電流密度後(1~12A/dm²)所生成的陽極膜顏色,其中,當電流密度為1~2A/dm²時陽極膜為透明顏色、當電流密度為3~5A/dm²時陽極膜為棕色顏色、當電流密度為6~7A/dm²時陽極膜為深褐色顏色、當電流密度為9~10A/dm²時陽極膜為黑色顏色、當電流密度為11~12A/dm²時陽極膜為黑色顏色,但是有局部白色燒毀痕跡。





圖一、6061 鋁合金表面陽極處理與圖案化步驟 圖二、6061 鋁合金陽極處理實際照片;電流密度:1~12A/dm²、50cm²、22 ℃、20min

## 三、 成果自評:

1	. 請就職型內容與原計畫相符程度、達成預期目標情況作一綜合評估
	達成目標
	一未達成目標(請說明,以100字為限)
	□執行失敗
	□因故執行中斷
	□其他原因
	說明:
	北江山县上上田上的小州市以及土上市社市山及建立。
2	. 執行計畫之成果在學術期刊發表或申請專利等情形:

論文:□已發表1_篇;■未發表之文稿;□撰寫中;□無
專利:□已獲得件;□申請中;□無
其他:(以100字為限) 附件1(未發表之文稿)

# 3. 其他:

4. 請依學術成就、技術創新、社會影響及在地發展等方面,評估執行成果之學 術或應用價值(簡要敘述成果所代表之意義、價值、影響或進一步發展之可 能性)(以500字為限)

本校發展特色為整合智慧橘綠科技,透過「厚實 i²GO 研究能量」、「提升國際化」及「深耕在地特色文化」等三大構面,提升國際競爭力。相對於課堂式的學理課程,本校學生對於實際動手操作實驗課程或參與研究計畫實作顯得更有興趣,本研究計畫也將提供給學生實際參與陽極處理設備的建置過程、陽極處理成品設計與製作、首先讓學生在實驗過程中獲得興趣再鼓勵學生深入探索陽極處理的理論。本計畫之另一目標為培養學生深入瞭解陽極處理技術,使學生畢業之後能夠應用所學。因為裝飾性陽極處理的競爭激烈,各陽極處理大般也積極的開發新市場,使陽極處理製程能順利進入下一代產品的關鍵製程,陽極處理一旦有了新的目標性產品,預計又將成為各製造業爭食的一塊大餅。

Surface Hardening of AISI 1020 Steel by Thermal Spray and Anodization

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#### Abstract

In this paper, an anodic film was developed on AISI 1020 steel through anodization. This anodic film can effectively improve the color, hardness, electrical insulation, and corrosion resistance of the steel surface. The fabrication process includes spraying an aluminum layer on the AISI 1020 surface by arc thermal spray method before anodization to form the anodic oxide film. This study also focuses on the effects of the anodic current density on the quality of the sprayed aluminum anodic film, including the microstructure, film thickness, hardness, surface roughness, and coloring. The sprayed aluminum film had a thickness of 400 µm and a porous microstructure, but after hot rolling, pores in the film were largely eliminated and the thickness was reduced to about 321 µm, a thickness reduction of 19.75%. The hot rolled aluminum film is embedded into grooves on the surface of the carbon steel to strengthen the adhesion of the aluminum

film on the surface. In this study, we suggest mild anodization parameters for a good quality anodic film as follows: anodic voltage of 20 V, a current density of  $3.5 \, \text{A/dm}^2$  at  $22 \, ^{\circ}\text{C}$  for 1h.

#### Keywords:

AISI 1020, Thermal spray, Anodization, Hardness, Surface, Microstructure

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#### 1. Introduction

Carbon steel is the earliest- and most-used basic material in modern industry. At present, carbon steel accounts for about 80% of the total steel output in the word. However, this type of steel often has limits, such as poor corrosion resistance, poor electrical insulation, low hardness, and colorlessness, which exclude it from special engineering applications. To improve the characteristics of the steel, two popular industrial technologies, namely, mass-production by thermal spraying [1, 2] and anodization technology [3, 4], can be combined to increase the application value of carbon steel surfaces. The common coating technologies, such as paint, vapor deposition, electro-deposition, and electrophoresis, provide only a thin film on the target substrate. However, thermal spraying can efficiently provide a thick protective film on the target substrate [5, 6]. In the main thermal spray process, molten or semi-molten metal particles are propelled toward the substrate by a stream of air, thus creating layer-by-layer deposition, until the required thickness of the coating is achieved [7].

2

Passive corrosion protection can be provided by anodic film. The anodic layer serves as a barrier to degradation of the metal surface and prevents contact between aggressive electrolytes and the underlying metal substrate [8]. Due to the porous structure of anodized film, a subsequent sealing treatment step is required. A beautiful, multi-colored dense film can be achieved with anodic film on modern products. This study examined in depth the effects of current density on the quality of sprayed aluminum after anodization, including the microstructure, film thickness, hardness, and surface roughness.

#### 2. Experimental procedure

Mild anodization film was formed on the AISI 1020 steel by thermal spraying and anodization in the present study. The experimental procedure can be primarily categorized into six stages: (a) Deposition of Al film on AISI 1020: depositing aluminum film with 400 μm thickness by arc thermal spray (30 min) on AISI 1020 steel (size of φ 2 inch and 5 mm thickness). (b) Densification of sprayed film: hot rolling at 100-200 °C to decrease the thickness of the sprayed film from 400 μm to 321 μm. (c) Pre-treatment for anodization: sample annealing (120 °C, 1h) in air furnace, mechanical grinding to remove any obvious scratches (#240, #600, and #1200 SiC sandpapers), and polishing of surfaces (1 μm Al<sub>2</sub>O<sub>3</sub> polishing powder). (d) Mild anodization (10 vol.% H<sub>2</sub>SO<sub>4</sub>, 20 V, 3.5 A/dm<sup>2</sup> at 22 °C for 1h for the substrate to form an anti-corrosion film with high hardness surface. (e) surface coloring after dyeing, (e) Water sealing to increase anodic film hardness and anti-corrosion property by water sealing (H<sub>2</sub>O, 100 °C, 40 min). (f) Mechanical polishing to improve anodic film surface (0.3 μm Al<sub>2</sub>O<sub>3</sub> polishing powder).

#### 3. Results and discussion

Figure 1 shows images of thermally sprayed Al on AISI 1020 substrate and form a coloring film after anodization

process. As shown in Fig. 1(a) a rust is on AISI 1020 surface with surface roughness values of  $R_a$  1.26  $\mu$ m. Figure. 1(b) AISI 1020 surface is clean and uniform surface roughness of 3.26  $\mu$ m through sand-blasting, Fig. 1(c) a thermal sprayed Al film of 400  $\mu$ m thickness is on AISI 1020 presented a porous structure with a large number of pores and 5.25  $\mu$ m surface roughness, Figure 1(d) the Al film after densification by hot rolling for future anodization. The film thickness was decreased from 400  $\mu$ m to 321  $\mu$ m, a reduction of 19.75%, and the surface roughness decreased from 5.25  $\mu$ m to 1.84  $\mu$ m, a reduction of 65 %. Figure 1(e) Anodic film with surface roughness of 0.25  $\mu$ m is on the thermal spray Al after mild anodization. Figure 1(f) Anodic film presented smooth surface roughness 0.11  $\mu$ m after polishing. Figures 1 (g), (h), and (i) coloring AISI 1020 surfaces are formed when the anodization film after dying. The anodization film improves the anticorrosion, coloring, anti-wear, insulation properties on AISI 1020. Also, the AISI 1020 surface hardness was increased to 350 HV<sub>0.55</sub>, as compared to the 120 HV<sub>0.11</sub> of AISI 1020.

In this study, the thermally sprayed Al film formed a hard and anti-corrosion oxide film on the carbon steel. Figure 2 (a) shows images of a 20 µm anodic film and a 321 µm thermal spray Al film on AISI 1020, (b) thermal spray Al film embedded into AISI 1020 surface, (c) the anode film acts as a high hardness and corrosion resistance film to protect the steel surface. The results showed that the rolled film could indeed block the electrolyte from penetrating to the AISI 1020 surface during anodization.

Because thermal spray Al has lower density and a lot of pores in the spray film compares to forging Al. and, defects or holes are between the steel substrate and spray film interface. The pores, defects, and holes make the electrolyte to penetrate from the sprayed Al film to the surface of the substrate steel during anodization and fail anodization. In order to make a high quality anodization film through thermally sprayed Al film the pre-treatment of hot rolling is needed and

the anodic parameters, including electrolyte composition, electrolyte temperature, anodic voltage, anodic current density, and anodic time, should be controlled well. In the anodization results we found a suitable anodization parameters of an anodic voltage of 20 V, a current density of 3.5 A/dm<sup>2</sup> at 22 °C for 1h for thermal spray Al.

Figure 3 showed optical images of interface between thermal spray Al and AISI 1020. (a) There are lot of pores (point 1) in the thermal layer and large holes (point 2) presented in the interface when the sample without hot rolling. (b) The pores (point 3) in the thermal layer were reduced and holes (point 4) size in the interface were getting smaller after 100 °C hot rolling. (c) The pores (point 5) and holes (point 6) are continuing reduced after 150 °C hot rolling. And, (d) The thermal spray film presented to denser (point 7) and the thermal spray film embedded (point 8) into AISI 1020 surface after 200 °C hot rolling by comparison before hot rolling.

#### 4. Conclusions

This novel and simple method offers a cost-effective approach to forming anodic film on an AISI 1020 surface. The key point is hot rolling, which can densify the thermally sprayed film on the steel surface and block the electrolytes from penetrating to the steel surface during the anodization process. This anodic film can effectively improve the surface properties of carbon steel, specifically, its corrosion resistance, surface hardness (from 120 HV to 350 HV), wear resistance, color, and electrical insulation. From the research results, we also found the anodization conditions for producing a high quality anodic film under 20 V, a current density of 3.5 A/dm² at 22 °C, 1h conditions. Combining thermal spraying, rolling, and anodization methods that can improve the surface of carbon steel. This feature can make carbon steel more commercially valuable for electromechanical components, medical equipment parts, or public engineering products.

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#### References

- [1] D.M. Kim, Y.S. Oh, S. Kim, H.T. Kim, D.S. Lim, S.M. Lee Thin Solid Films 2011; 519:6698-6702.
- [2] K. Miwaa, N. Takada J. Vac. Sci. Technol. A 2009; 27:831-835.
- [3] C.C. Chen, S.F. Chang, Z.P. Luo Materials Letters, 2013; 112: 190-193.
- [4] Wu Yuping, Lin Pinghua, Chu Chenglin, Wang Zehua, Cao Ming, Hu Junhu Materials Letters 2007; 61:1867-1872.
- [5] Mohammed Thalib Basha, Akella Srikanth, B. Venkateshwarlu Materials Today: Proceedings 2020; 22:1554-1562.
- [6] Berenika Syrek-Gerstenkorn, Shiladitya Paul, Alison J Davenport Coatings 2020; 10:267, 19 pages.
- [7] Xiaotao Luo, Gregory M. Smith1, Sanjay Sampath J Therm Spray Tech 2018; 27:296-307.
- [8] Cavezza F., Boehm M., Terryn H., Hauffman T. A Metals 2020; 10:730, 32 pages.

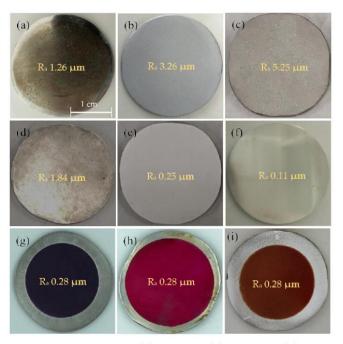


Figure 1 Coloring AISI 1020 surface formation through (a)AISI surface, (b)sand-blasting, (c) thermal spray, (d) hot rolling, (e) anodization, (f) polishing, (g), (h), and (i) dyeing process.

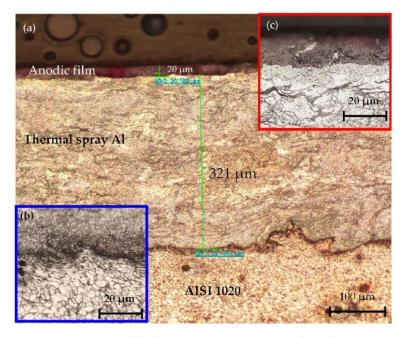


Figure 2 a hard and anti-corrosion surface of AISI 1020 steel; (a) anodic film and a thermal spray Al film on AISI 1020, (b) interface between thermal spray Al and AISI 1020, (c) interface between anodization film and thermal spray Al.

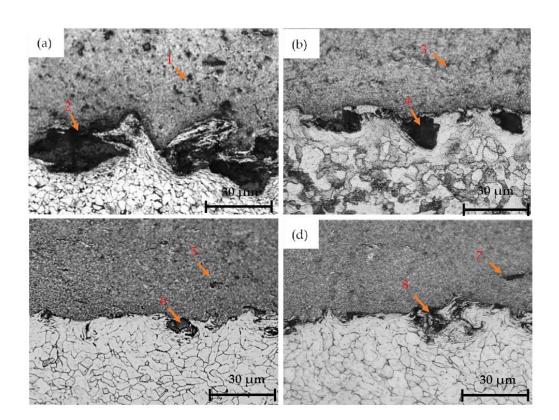


Figure 3 interface between thermal spray Al and AISI 1020, (a) without hot rolling, (b) after 100  $^{\circ}$ C, (b) after 150  $^{\circ}$ C, and (c) after 200  $^{\circ}$ C hot rolling.